

FORM PTO-1390 (Modified) (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER 4004-033-30	
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR <div style="font-size: 1.5em; font-weight: bold; text-align: center;">10/088295</div>	
INTERNATIONAL APPLICATION NO. PCT/EP00/08948		INTERNATIONAL FILING DATE 11 SEPTEMBER 2000		PRIORITY DATE CLAIMED 16 SEPTEMBER 1999	
TITLE OF INVENTION <div style="text-align: center; font-weight: bold; margin-top: 10px;">TRANSPARENT FIRE BREAK GLAZING</div>					
APPLICANT(S) FOR DO/EO/US <div style="text-align: center; font-weight: bold; margin-top: 10px;">GOELFF, Pierre, et al.</div>					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
<ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below. 4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c) (2)) <ol style="list-style-type: none"> a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). 7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input checked="" type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)). 11. <input type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409). 12. <input type="checkbox"/> A copy of the International Search Report (PCT/ISA/210). 					
Items 13 to 20 below concern document(s) or information included:					
<ol style="list-style-type: none"> 13. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 14. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 15. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 16. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 17. <input type="checkbox"/> A substitute specification. 18. <input type="checkbox"/> A change of power of attorney and/or address letter. 19. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 20. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 21. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 22. <input type="checkbox"/> Certificate of Mailing by Express Mail 23. <input checked="" type="checkbox"/> Other items or information: <div style="margin-top: 10px;"> <p>A copy of the International Application as published (WO 01/19608) , with copies of the International Search Report in English and French, and an English-language abstract</p> <p>A White Advance Serial Number Postcard</p> </div> 					

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.53) <div style="font-size: 1.5em; font-weight: bold;">10/088295</div>	INTERNATIONAL APPLICATION NO. <div style="font-weight: bold;">PCT/EP00/08948</div>	ATTORNEY'S DOCKET NUMBER <div style="font-weight: bold;">4004-033-30</div>
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24. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO	\$1040.00
<input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO	\$890.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO	\$740.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)	\$710.00
<input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)	\$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

CALCULATIONS PTO USE ONLY

Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)). <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 				\$130.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	38 - 20 =	18	x \$18.00	\$324.00	
Independent claims	2 - 3 =	0	x \$84.00	\$0.00	
Multiple Dependent Claims (check if applicable).				<input type="checkbox"/>	\$0.00
TOTAL OF ABOVE CALCULATIONS				=	\$1,344.00
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27). The fees indicated above are reduced by 1/2.				\$0.00	
SUBTOTAL				=	\$1,344.00
Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30 				+	\$0.00
TOTAL NATIONAL FEE				=	\$1,344.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input type="checkbox"/>				\$0.00	
TOTAL FEES ENCLOSED				=	\$1,344.00
				Amount to be refunded	\$
				charged	\$

a. ☒ A check in the amount of 1,344.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-1442 A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Supervisor, Patent Prosecution Services
PIPER MARBURY RUDNICK & WOLFE, LLP
 1200 Nineteenth Street, NW
 Washington, DC 20036-2412
 US

SIGNATURE

SCHNEIDER, Jerold I.
 NAME

24,765
 REGISTRATION NUMBER

15 MARCH 2002
 DATE

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Surcharge of **\$130.00** for furnishing the oath or declaration later than ☐ 20 ☒ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).

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CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
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Multiple Dependent Claims (check if applicable).

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TOTAL OF ABOVE CALCULATIONS =

\$1,344.00

- ☐ Applicant claims small entity status. See 37 CFR 1.27). The fees indicated above are reduced by 1/2.

\$0.00

SUBTOTAL =

\$1,344.00

Processing fee of **\$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)).

\$0.00

TOTAL NATIONAL FEE

\$1,344.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable).

\$0.00

TOTAL FEES ENCLOSED

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A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-1442 A duplicate copy of this sheet is enclosed.
- d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

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Supervisor, Patent Prosecution Services
PIPER MARBURY RUDNICK & WOLFE, LLP
1200 Nineteenth Street, NW
Washington, DC 20036-2412
US

SIGNATURE

SCHNEIDER, Jerold I.

NAME _____

24,765

REGISTRATION NUMBER

15 MARCH 2002

DATE _____

10/088295

IC10 Rec'd PCT/PTO 15 MAR 2002

DOCKET NO. 4004-033-30

**PATENT COOPERATION TREATY
IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)**

#3/a

In re Application of: GOELFF, Pierre, et al.
Int'l Application No.: PCT/EP00/08948
Int'l Filing Date: 11 September 2000
Priority Date: 16 September 1999
U.S. Application No.: New U.S. National Stage Application
For: **TRANSPARENT FIRE BREAK GLAZING**

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS
Box PCT
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, and with reference to the accompanying English translation, please amend this application as follows:

IN THE SPECIFICATION

Page 1, prior to line 1, insert the section heading followed by the text as follows:

- - CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority from PCT Application No. PCT/EP00/08948 filed 11 September 2000, which is based upon and claims priority from Belgium Application No. 9900619 filed 16 September 1999, both of which are hereby incorporated by reference.

BACKGROUND OF THE INVENTION - -

Page 2, between lines 23 and 24 insert the section heading:

- - SUMMARY OF THE INVENTION - -

Page 7, between lines 24 and 25 insert the section heading:

- - DETAILED DESCRIPTION - -

IN THE CLAIMS

Cancel claims 1 through 13 without prejudice or disclaimer and insert the following new claims 14 through 38.

- - 14. Transparent fireproof glazing comprising at least two glass sheets and an intumescent layer of material being located between the glass sheets, the layer containing a phosphate-based compound and a substance from the group consisting of pyrogenous silica and a mixture of pyrogenous silica and alumina.

15. Glazing according to Claim 14, wherein the phosphate-based compound is a hydrogen phosphate of X, where X is selected from the group consisting of Mg, Ca and Al.

16. Glazing according to Claim 15, wherein the phosphate-based compound has an atomic ratio P/X greater than 2.0.

17. Glazing according to Claim 14, wherein the intumescent layer further includes at least one of :

- (a) a salt of aluminum, or
- (b) a hydroxide of aluminum.

18. Glazing according to Claim 17, wherein X is Mg and the atomic ratio of Al/Mg is in the range of between 0.1 to 0.4.
19. Glazing according to Claim 14, and further including at least one of the following features (a) through (c):
- (a) the silica in the intumescent layer is composed of particles having a grain size in the range of between 7 and 40 nanometers;
 - (b) the quantity of silica in the intumescent layer is between 1 and 10 % by weight of the intumescent layer;
 - (c) the intumescent layer includes water in the amount of between 18 to 40% by weight of such layer.
20. Glazing according to Claim 19, and including at least two of the features (a) through (c).
21. Glazing according to Claim 19, and including all of the features (a) through (c).
22. Glazing according to Claim 14, wherein the quantity of silica in the intumescent layer is between 2 and 6 % by weight.
23. Glazing according to Claim 14, wherein the intumescent layer includes water in the amount of between 30 to 35% by weight of such layer.
24. Glazing according to Claim 14, wherein the intumescent layer includes water in an amount which is adjusted so that the refractive index of the intumescent layer approximates the

refractive index of the silica contained therein.

25. Glazing according to Claim 14, wherein the intumescent layer includes an additive so that the refractive index of the intumescent layer approximates the refractive index of the silica contained therein.

26. Glazing according to Claim 25, wherein the additive is present in an amount not exceeding 15% by weight of the intumescent layer.

27. Glazing according to Claim 25, wherein the additive is selected from the group consisting of glycerol, DMSO and ethylene glycol.

28. Glazing according to claim 25, wherein the additive has a higher index of refraction than the index of refraction of the phosphate-based compound.

29. Glazing according to Claim 14, wherein the intumescent layer includes a water content and a silica content such that the intumescent layer is sufficiently fluid to flow between the glass sheets and further forms a gel therebetween in approximately 24 hours.

30. A method of forming a fireproof glazing including at least two glass sheets with an intumescent layer therebetween, the intumescent layer having sufficient fluidity to flow between the glass sheets and to thereafter form a gel therebetween, comprising the steps of:

- (a) providing a phosphate-based solution having a first index of refraction;
- (b) mixing a pyrogenous silica having a second index of refraction with said

phosphate-based solution; and

(c) adjusting as necessary the first index of refraction to approximate the second index of refraction such that the intumescent layer flows between the glass sheets and thereafter forms a gel as aforesaid.

31. The method according to Claim 30, wherein step (c) is at least one of the following:

(d) adjusting the amount of water in the phosphate-based solution;

(e) providing an additive to change the first index of refraction.

32. The method according to Claim 31, and including both steps (d) and (e).

33. The method according to Claim 30, wherein the amount of said pyrogenous silica is between about 1 and 10% by weight of the intumescent layer.

34. The method according to Claim 30, wherein the phosphate-based solution has an atomic ratio P/X greater than 2, and where X is selected from the group consisting of Mg, Ca and Al.

35. The method according to Claim 30, wherein the intumescent layer further includes a crystal formation retardant which at least retards, if not prevents, the formation of silica crystals.

36. The method according to Claim 25, wherein the phosphate-based solution includes phosphate of magnesium and the crystal formation retardant includes aluminum present in an atomic ratio Al/Mg in the range of between 0.05 and 0.4.

37. The method according to Claim 31, wherein the additive is present in an amount not exceeding 20% by weight of the intumescent layer.

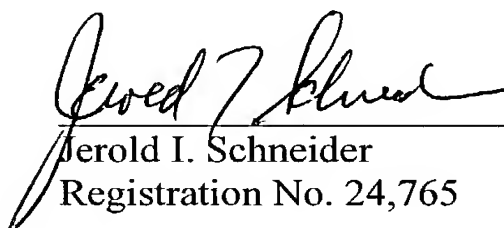
38. The method according to Claim 37, wherein the additive is selected from the group consisting of glycerol, DMSO and ethylene glycol. - -

REMARKS

Claims 14 through 38 are pending. The specification has been amended to place the application in more conventional U.S. format, and the claims have been amended to eliminate multiple dependency and present claims of varying scope. Favorable consideration is solicited. Should the Examiner be of the opinion that a personal interview will expedite the prosecution of this application, the Examiner is encouraged to call Applicants' attorney at the following telephone number.

Respectfully submitted,

PIPER MARBURY RUDNICK & WOLFE LLP


Gerald I. Schneider
Registration No. 24,765

1200 Nineteenth Street, N.W.
Washington, D.C. 20036-2412
Telephone No. (202) 861-3900
Facsimile No. (202) 223-2085

TRANSPARENT FIRE BREAK GLAZING

The invention relates to fire break glazing units comprising an intumescent material with improved properties.

Fire break glazing of this type can lead to the use of different materials. The choice of intumescent materials has a significant influence not only on the thermal characteristics of the glazing but also on the production techniques of such glazing which in particular are one of the significant factors in their cost price.

The products demanded by users must have ever more effective properties. In particular, glazing units must be fire resistant for an increasingly long period in increasingly more severe temperature conditions. To meet these new demands, the manufacturers of such glazing units have proposed in particular the use of new intumescent materials with better resistance to very high temperatures. When evaluating the resistance of these materials, an important factor is the temperature at which the intumescent material melts or breaks down after formation of the foam in fire test cycles. This transformation in the material is associated with the loss of its insulating properties. If the most usual intumescent materials, those based on hydrated alkaline silicates, transform at a temperature in the order of 400 to 500°C, other materials enable 700°C to be reached and exceeded.

Of the materials, which lead to foams resistant to the most elevated temperatures, compositions containing phosphates are those which appear to respond best to these new demands. Materials of this type have been the subject of patent publications, in particular WO 99/19422 in the name of the Applicant. Apart from their increased thermal resistance, intumescent materials of this type differ from the most usually used silicate-based materials by their rheological properties and their conditions of use resulting from these. In particular, these phosphate-based compositions gel in very specific conditions, and do not always exhibit the required stability in thermal shock tests. The main difficulty is the tendency of phosphate-based intumescent materials to creep under the effect of the heat at temperatures lower than those at which the foam forms.

It is understood that the efficiency of fire break glazing requires that each of the constituents properly plays its role. Intumescent material contributes in various ways. It absorbs part of the heat received by the glazing by vaporizing the hydrating water it contains. It also forms a screen for thermal conduction and radiation via the opaque foam it forms during evaporation of this same water. Finally, the material holds the glass sheets in position even after they have been shattered by the thermal shock. If the material is removed by creep before causing the formation of foam, it cannot play any of these roles which are essential to the favorable functioning of fire break glazing.

In order to increase the fire behavior of glazing units, one measure currently used is to increase the number of glass sheets and intumescent interlayers. The principle is to successively oppose factors which accordingly slow down the final destruction of the glazing. In the case of an assembly comprising several layers of intumescent phosphate-based material, the difficulty may come from inadequate creep resistance at the temperatures preceding expansion. In fact, if during the fire test the thermal shock enables the first intumescent layer to expand properly, the temperature of the following layers "protected" by this first thermal barrier increases more slowly. If the intumescent material is not sufficiently resistant to creep at high temperature, the subsequent layers are not able to contribute satisfactorily.

Therefore, the inventors have sought to find improved intumescent materials which have a very high foam transformation temperature and at the same time are creep resistant at the intermediate temperatures preceding formation of the foam.

The inventors have shown that intumescent materials intended to form part of the composition of fire break glazing based on phosphates and comprising appropriate additives could meet this objective. The materials according to the invention contain as additive a very fine powder of pyrogenous silica or a mixture of pyrogenous silica and aluminum oxide.

The inventors have shown interest especially in incorporating these pyrogenous silicas possibly mixed with aluminum oxide to modify the properties of the intumescent materials formed from hydrated hydrogen phosphates of magnesium, calcium or aluminum, such as those described in the above-cited publication.

Powders of pyrogenous silica or a mixture of pyrogenous silica and aluminum oxide are commercially available in particular as fluidizing agent for powders, as filler in plastic materials or to serve as thickening agents in various products such as paints, cosmetic products etc.

5 The use of thickening agents in intumescent materials to modify their rheological properties has already been proposed. To maintain the transparent character of glazing it would therefore appear necessary to choose products which are not only compatible with the other constituents of the material, but also are soluble in this material.

10 The choice of suitable products is all the more sensitive since phosphate-based materials are very aggressive. Alkaline phosphates result in compositions with very acid pH values which are incompatible with the most usual thickening agents. Thickening agents previously proposed for these intumescent layers were, for example, hydroxyethyl celluloses, polyvinyl alcohols, natural
15 rubbers etc. These have otherwise proved to be poorly suited to the effective conditions of use of the products under consideration. In particular, they do not further the formation of foam, which determines the essential properties of the glazing in its role as firebreak.

 The inventors have shown that the incorporation of pyrogenous silica
20 or a mixture of pyrogenous silica and aluminum oxide allowed modification of the rheological properties of the intumescent materials of fire break glazing with high thermal decomposition temperatures by using non-soluble thickening agents with good resistance to the pH conditions, and when used appropriately result in intumescent layers which also meet optical requirements: transparency, absence of
25 air bubbles, crystals, and generally without any fault detrimental to the qualities of the final glazing.

 Pyrogenous silicas are formed from particles of very small dimensions. Their average grain size lies between 7 and 40 nanometers, depending on type. The presence of hydroxyl groups on their surface provides them with a
30 marked hydrophilic character. Pyrogenous silica particles are not changed in a highly acid medium. The introduction of this additive into the compositions according to the

invention leads to the dispersion of particles into the intumescent material without dissolution in this.

The quantity of pyrogenous silica is advantageously in the range of between 1 and 10% by weight of the composition, preferably between 2 and 6%.

5 For various reasons, it is preferable to introduce the lowest possible effective quantity. The presence of particles in suspension generates a certain diffusion of light. Therefore it is desirable to limit the quantity of silica. However, to obtain the effect of a creep resistant gel, a minimum quantity of silica is clearly necessary. The most suitable proportions must be determined according to the case,
10 taking into consideration many contributing factors, especially the quantity of water in the produced composition.

For the applications in question, the water content in the intumescent material is fixed to meet various, sometimes contradictory demands.

15 Firstly, the water content must properly assure the formation of foam when the glazing is exposed to fire. For this, a certain quantity of water is obviously required which will allow expansion of the material when it evaporates. To enable the foam to exhibit good resistance to convection and radiation, it is necessary that it is formed from a large quantity of uniformly distributed pores. Experience shows that to provide good expansion, the quantity of water must be precisely selected. It
20 appears that for too large a quantity of water, the air bubbles, which initially appear in large number during expansion, become too large and cause disorderly coalescence. The result is a foam with pores of very irregular dimension, which exhibits a poorer thermal resistance. Secondly, the water content significantly influences the stability of the optical and mechanical properties of the intumescent
25 layer. The lower the content, the greater the risk of crystals forming. A very low water content may also lead to ageing faults in the layer. Cracks and fissures can appear in the course of time. Conversely, a high water content prevents the formation of crystals, but, as we will see below, contributes to the increase in light diffused by the silica particles.

30 The water content also has an influence on the conditions in which the preparation of the intumescent layer is conducted. Once again, contradictory conditions must be met. The preparation of the material and its formation are

facilitated by a high water content, which also results in a low viscosity. In particular, as the material is fluid, the glazing can be produced by the material flowing directly between the glass sheets, which enables economic formation of a sheet of intumescent material and of the subsequent assembly of this with glass sheets.

5 However, if the initial fluidity has the advantages, which have just been indicated, the material must ultimately coagulate into a gel, which is more difficult to achieve when the initial water content is higher. If the water content of the material is too high, for example, because of the conditions determined by its preparation, then it becomes necessary to proceed to a more or less lengthy and delicate drying step to

10 achieve the degrees of hydration and firmness of the desired gel. As far as possible, the aim is to prepare a composition of intumescent material which does not require drying or in which the drying step is as limited as possible. The examples will show more precisely how to direct the choices allowing these different demands to be met.

In general, the quantity of water contained in the intumescent material

15 of the glazing is not less than 18%, and not more than 40% to meet the conditions of expansion leading to the formation of a homogeneous foam. To ensure that the intumescent layer practically does not show any diffusion of light (preferably less than 1%), without having recourse to any possible means allowing the correction of what appears as a fault and which will be discussed below, the water content is

20 advantageously at most equal to about 35%, preferably 30%. Finally, to direct the composition into a state of fluidity, which allows it to flow easily between glass sheets, the water content is advantageously at least 30%. Consequently, it is difficult to meet all the conditions simultaneously without having recourse to other means, which are also the subject of the invention.

25 The introduction of pyrogenous silica according to the invention, as indicated above, is accompanied by a dispersion of particles, which leads to a certain diffusion of light. Even if this diffusion remains limited, it is preferable in order to improve the optical quality of the glazing to further reduce it, even to eliminate it completely. In all cases, to give satisfaction, the transparent glazing according to the

30 invention must not have a total diffusion greater than 3%, and preferably this diffusion must be at most equal to 1%.

We have stated that a low water content would be preferable to restrict the diffusion. The inventors have further shown that it was possible to cause the diffusion to disappear by adjusting the water content. The reason for this is as follows. The introduced silica particles have a refractive index of slightly less than 1.5. The base compositions of the solution intended to form the intumescent layer, with water contents corresponding to the modes of preparation described in particular in the WO patent application referred to, normally have refractive indexes which are different from those of the particles. These compositions normally have a lower refractive index in the order of 1.4. The difference between the indexes is the cause of the diffusion. The inventors have shown that it was possible to modify the index of these compositions until that of the particles was reached by changing the water content. A reduction in the water content, as indicated in the examples, allows an increase in the index, if necessary until that of the particles is equaled.

In any case, it is possible according to the invention to reduce the diffusion without changing the water content by introducing additives into the composition, which allow the refractive index of the composition to be modified. The choice of these additives and their content in the composition, as above, aims at directing the solution to an index, which is as close as possible to that of the silica of the particles.

As a general rule, additives allowing the refractive index to be corrected are products, which may be mixed, in the base solution and their own index is different from that of the solution. Naturally, the additives must not have a negative influence on the properties of the intumescent layer.

For very acid phosphate-based compositions, the additives correcting the index must be stable in this medium. They must have a higher index than that of the base composition, and preferably even higher than that of the pyrogenous silica. The following compounds are suitable candidates, for example: 4-acetyl-morpholine (n 1.483), sulphonal (n 1.484), furfuryl alcohol (n 1.4845), (2-amino-ethylamino)-2-ethanol (n 1.4861), ethyl pyridine-3-carboxylate (n 1.504), (piperazinyl-1-)-2-ethanol (n 1.5052), 3-phenyl propionic acid (n 1.5106), 2-phenyl butyric acid (n 1.515), N-methylbenzylamine (n 1.5235), 1-phenylethanol (n 1.5265), (pyridyl-2)-2-ethanol (n 1.5368), benzylic acid (n 1.5396), allyl-2-phenol (n 1.544), 2-chlorophenol (n

1.5579), 2-anilinoethanol (n 1.5593), 2-chlorophenol (n 1.5793). The preferred additives are glycerol and dimethyl sulphoxide (DMSO). These compounds can represent up to 15% by weight of the total material of the intumescent layer, but preferably amount to less than 10%.

5 The base materials forming the intumescent layer are phosphates of magnesium, calcium or aluminum, with a P/Mg (or Ca or Al) ratio higher than 2. The material is no longer sufficiently transparent at values for this ratio below the indicated limit. It takes on a milky appearance which is not normally wanted for glazing, and which results from the precipitation of insoluble phosphates when the
10 medium is not sufficiently acid.

 In the final product the intumescent layer must retain its properties, in particular transparency, for so long as the glazing is not subjected to the fire test. It is also necessary that in usual ambient conditions the intumescent gel does not develop disruptive crystallization, or does not enable the formation of air bubbles or cracks in
15 sufficient quantity to modify its appearance in a totally unacceptable manner. In particular, depending on the water content of the gel, it may be advantageous to add a salt or hydroxide of aluminum to the composition. The presence of these compounds, especially the salts AlCl_3 or AlNO_3 , prevents or substantially slows down the formation of crystals in the intumescent layer incorporated into the glazing. The
20 preferred proportions for this additional compound of aluminum are advantageously such that the atomic ratio Al/Mg is in the range of between 0.05 and 0.4.

 The preparation of the intumescent layer forming part of the composition of the glazing according to the invention is outlined in detail in the following description relating to practical examples.

25 The test solutions are formed from hydrated magnesium dihydrogen phosphate. The solution is supplemented by a constituent of aluminum, depending on the case. In order to form the base solution, an 85% aqueous solution of phosphoric acid is used. Magnesium tetrahydroxycarbonate ($4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) is added.

30 The molar ratio P/Mg is always higher than 2. With lower values the solution causes the formation of poorly soluble $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ which precipitates. A

typical base solution is formed with a P/Mg ratio of 2.24. Based on the compounds indicated, the prepared solution contains 57% water. Its pH is about 1.6 to 1.7.

The magnesium hydroxycarbonate is dissolved by intense agitation. Dissolution is slow. It is assisted to a limited degree by an increase in temperature. 5 Once dissolution has been completed, the solution is filtered to eliminate any undissolved particles or crystals remaining which as such would constitute faults in the final layer.

To attain the various concentrations of water forming the subject of these tests, the base composition, initially formed as a gel, is dried in a ventilated 10 drying oven. As already known, the drying can be performed in a microwave heating chamber. When performing these tests in the case of the compositions according to the invention including silica, the formation of the gel precedes the drying which modifies the concentration of water. This procedure prevents any irregularity from forming in the structure of the final gel, i.e. such irregularities as folds, grains or 15 cracks, faults that are observed when in the rest of the operations drying precedes gelling.

The problems of conducting drying operations are well known. It is a matter of evacuating part of the water from the intumescent layer being formed, while retaining perfect homogeneity in the product. In particular, the formation of air 20 bubbles or crystals must be prevented during this treatment, and progressive drying in a ventilated oven is preferred for this. Starting from a gel, drying is necessarily slow, in particular so as to avoid the formation of a "skin" on the surface in contact with the atmosphere of the drying oven. This very restrictive drying operation is reduced as far as possible, and even omitted in the industrial production of the 25 products according to the invention. However, this type of drying has been used for the requirements of these tests in order to respond to the necessity of determining the behavior of products studied in various conditions.

Without adding pyrogenous silica, the drying necessary for forming a suitable intumescent layer can be considerable. Without silica, experience has shown 30 that a water content lying between 20 and 30% by weight of the layer is required to obtain a layer, which forms an adequate foam. The difficulty with these values, apart from the length of treatment, is to arrive at a favorably homogeneous intumescent

layer, which retains the desired optical quality. The addition of pyrogenous silica enables products containing substantially higher contents of water to be worked with, and thus to at least partially eliminate these difficulties.

5 The addition of silica is conducted on the solution of phosphates, which has been prepared and filtered as indicated above. Incorporation of the silica powder into the solution requires vigorous agitation, since the particles tend to form more or less voluminous accumulations. The perfectly uniform dispersion of particles is all the more difficult when the solution has a lower water content and is therefore more viscous. An excess amount of silica in relation to the useful quantity
10 is therefore not only superfluous but may also complicate the process of obtaining a homogeneous composition.

The silica used for the tests is that marketed by Degussa under the name "Aerosil 200". This is a powder having particles with an average grain size of 12 nanometers.

15 The quantity of powder introduced into the solution to obtain a gel depends on the water content. The nature of the powder and the manner in which it influences the modification of the solution mean that beyond a certain relatively low quantity, the addition of silica adds nothing to the rheological characteristics of the composition. In the tests, the added silica represented 3, 4 or 5% by weight of the
20 intumescent material.

The quantity of silica necessary to produce a suitable gel increases on first approximation with the water content of the intumescent material. Thus, it is possible to obtain a gel in 24 hours working from a solution of 57% water by adding 5% by weight of silica. With lower proportions of silica, e.g. 4%, and working from
25 the same solution, no gel is obtained even after a resting period of 7 days. With a solution with 37.5% water, an addition of 4% silica is sufficient to form a gel in less than 24 hours.

The above statements show that gel formation is not instantaneous, this being dependent on the nature of the constituents and on the way they react with
30 each other. The bonds configure slowly within the composition until the structural balance corresponding to the gel is reached.

The prepared compositions are introduced into test cells reproducing glazing conditions. The cells are formed from two 200x200 mm glass sheets, between which the intumescent layer is located.

5 The gels forming the intumescent layer containing different proportions of water are firstly tested for their creep resistance at temperatures of 50 and 80°C. With the cells placed in a vertical position, the behavior of the 2 mm thick intumescent layer at the indicated temperatures is observed. Tests were conducted with compositions containing 35, 38.5 and 42% water and 4 and 5% silica. No creep was observed at the indicated temperatures after 24 hours. By comparison, a gel of
10 the same composition but without silica cannot be maintained in these conditions in the cell. Creep is evident in the intumescent layer without silica even in a material with a water content less than that of the products according to the invention concerned in this test.

Therefore, the materials according to the invention subjected to the
15 fire test cycle are not degraded at the start of the cycle. They remain in position and are able to expand properly to form an insulating foam. Continuation of the cycle showed that the foam formed by expansion is resistant up to temperatures higher than 700°C. Decomposition of the foam occurs between 700 and 900°C. Fusion of the material results at about 1100°C.

20 The addition of pyrogenous silica, in other words of solid particles, to the solution of phosphates with 57% water prepared as indicated above, causes a certain light diffusion forming a bluish halo. If the water content is reduced, the refractive index of the composition of the intumescent material increases. To eliminate this halo, the water content must be brought to a level where the refractive
25 index approaches that of the silica particles. The refractive index in the case of Aerosil 200 is 1.46. Measurements of the index in relation to the water content show that the composition reaches this index with a water content in the order of 30%. With this value the composition of the material has a homogeneous refractive index. There is no further light diffusion.

30 It goes without saying that the water content allowing adjustment of the indexes of the solution and of the silica is dependent on the precise composition of the solution. Even if the variations in index are relatively insignificant with respect

to the composition, they lead to quite different water contents for "equalizing the indexes". The addition of aluminum chloride, for example, increases the index of the composition and enables the index of the silica to be reached with a higher water content, all other factors being equal. Conversely, an increase in the P/Mg ratio tends to reduce the index of the solution. As an indication, with a P/Mg ratio equal to 3 (the ratio of the reference solution being 2.24), the index for 30% water is about 1.44. It would have to be possible to prepare a composition containing less than 20% water to reach the index of the silica. This solution is not preferred either from the point of view of being able to prepare this composition by drying or with respect to the qualities of the resulting layer to form insulating foam.

In the last situation, it is possible to adjust the index of the composition by means of additives such as glycerol or DMSO so as to achieve an index close to that of the silica. When maintained in proportions not greater than 20%, these compounds and in particular glycerol do not appreciably modify the qualities of the intumescent material. It is desirable to limit the content to the indicated value. With higher contents, carbonization of these organic compounds during the fire test could increase thermal conduction of the foam formed and thus reduce the resistance of the glazing.

As an indication, the refractive index of a solution of phosphates (P/Mg 2.24 and Al/Mg 0.3) containing 4% silica and 37% water will be seen to go from 1.44 to almost 1.45 as a result of adding glycerol. The same composition with 40% water goes from 1.435 to 1.443 as a result of adding 10% DMSO.

The increase in the index has a direct influence on the optical quality of the intumescent layer. Even if the indexes of silica and the solution are not identical, the closeness allows the measured diffusion to be appreciably reduced. In the case of the examples of compositions indicated above, the layer without glycerol has a diffusion of 1.18%. The same layer containing glycerol only has a diffusion of 0.87%. Therefore, by combining the actions of the water content and the additives, it is possible to correct the effect of diffusion resulting from the presence of silica until acceptable values are achieved.

The compositions prepared according to the above indications have also been subjected to ageing analyses. The materials must retain properties, in

particular those of transparency and of absence of optical faults over very long periods of use. The most significant factor in this respect is the risk of crystals being formed in the transparent material.

5 The inventors have been able to verify that the presence of aluminum chloride or nitrate in the composition enables the formation of crystals to be prevented or considerably reduced. The addition of the aluminum compound is preferably performed before the introduction of silica and after filtration of the solution of phosphates.

10 The products prepared in these tests contain a quantity of aluminum ion such that the atomic ratio Al/Mg is 0.1, 0.2 and 0.3 respectively. The layers have a composition comprising 43% water and 5% silica. The P/Mg ratio remains 2.24. The test samples are observed for 24 hours and 7 days after preparation. The product with a low aluminum content (Al/Mg 0.1) exhibits a significant number of crystals from the first day. After a week, the same product has been colonized by crystals.
15 The second sample (Al/Mg 0.2) only exhibits a small number of crystals at one day and a more significant number after a week. Finally, the last sample (Al/Mg 0.3) does not contain any fault after 24 hours and a completely acceptable number of discernible crystals after a long ageing period. The presence of the aluminum compound therefore enables the appearance of undesirable crystallization faults to
20 be favorably controlled.

As indicated above, the compositions of intumescent materials according to the invention have the further advantage, if necessary, of facilitating the production of glazing units by allowing the composition to be introduced directly between the glass sheets of the glazing. By appropriate selection of the constituents
25 of this composition and the proportions thereof, it is possible to prepare a solution having rheological characteristics which, on the one hand, allow introduction of the composition in a sufficiently fluid form for it to flow freely and occupy the space between the glass sheets and, on the other hand, result in a properly stable gel with all the required thermal and optical characteristics within a few hours.

30 In the case of the compositions indicated above (P/Mg 2.24), the lowest water content which allows this flow between the sheets is about 35%. Compositions, which have at least this water content and which can be gelled by the

addition of silica can result in the formation of glazing units without any drying treatment. The products according to the invention, on the one hand, exhibit the advantage of a better thermal resistance and, on the other hand, with respect to some of them at least, they can be produced in the simplest conditions to be applied.

5 The glazing units according to the invention can be produced with all types of glass sheets used for these applications. These may be the most usual soda-lime glass sheets. Sheets of borosilicates or even glass ceramic sheets may also be included, wherein the qualities of low thermal expansion constitute a frequently sought advantage. The glass is most frequently white, but may also be solidly
10 colored. The thermal behavior of the glasses may be further improved by using thin layers having reflective properties in particular. In the same glazing layers of silicate gel can alternate with layers of phosphate gels.

 Use of the glazing units according to the invention permits all the standard types of assemblies. They may be used in single or multiple glazing units, in
15 mobile assemblies (doors, windows) or in fixed assemblies (wall elements, facades etc.), and generally in all traditional applications of fire break glazing units.

CLAIMS

1. Transparent fire-proof glazing comprising at least two glass sheets and an intumescent layer of material containing a phosphate-based compound, said layer being located between these two glass sheets, wherein the intumescent material comprises pyrogenous silica or a mixture of pyrogenous silica and alumina.
2. Glazing according to Claim 1, wherein the intumescent material is based on hydrogen phosphates of magnesium, calcium or aluminum.
3. Glazing according to Claim 1 or Claim 2, wherein the pyrogenous silica is composed of particles, the average grain size of which is in the range of between 7 and 40 nanometers.
4. Glazing according to any one of the preceding claims, wherein the quantity of pyrogenous silica in the intumescent layer is in the range of between 1 and 10% by weight of the composition forming this layer.
5. Glazing according to any one of the preceding claims, wherein the quantity of pyrogenous silica in the intumescent layer is in the range of between 2 and 6% by weight of the composition forming this layer.
6. Glazing according to any one of the preceding claims, wherein the composition of the intumescent layer contains from 18 to 40% by weight of water.
7. Glazing according to any one of the preceding claims, wherein the composition of the intumescent layer contains from 30 to 35% by weight of water.
8. Glazing according to Claim 4, 5 or 6, wherein the water content is adjusted so as to bring the refractive index of the composition to a value close to that of the pyrogenous silica, which it contains.
9. Glazing according to any one of the preceding claims, wherein the refractive index of the intumescent layer is brought close to that of the pyrogenous silica by adding glycerol, dimethyl sulphoxide or ethylene glycol in a proportion not exceeding 15% by weight of the composition of the intumescent layer.
10. Glazing according to any one of Claims 2 to 9, wherein the composition of the intumescent layer has an atomic ratio P/Mg (or Ca or Al) greater than 2.

11. Glazing according to any one of the preceding claims, wherein the composition of the intumescent layer additionally contains a salt or hydroxide of aluminum.

12. Glazing according to Claim 11, wherein the aluminum is present in such a quantity that the atomic ratio Al/Mg is in the range of between 0.1 and 1.

13. Glazing according to any one of the preceding claims, wherein the composition of the intumescent layer comprises a pyrogenous silica content and a water content such that, during preparation of the composition, this has a fluidity which permits it to be inserted between the sheets of the glazing, said composition forming a gel after approximately 24 hours.

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En ce qui concerne les codes à deux lettres et autres abrévia-
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(54) Title: TRANSPARENT FIRE BREAK GLAZING

(54) Titre: VITRAGE COUPE-FEU TRANSPARENT

(57) Abstract: The invention concerns transparent fire break glass panels. The inventive glazing comprises at least two glass sheets and an intumescent phosphate-based material layer, layer which is located between said two glass sheets. The intumescent material comprises pyrogenous silica or a mixture of pyrogenous silica and alumina which enables said material to be exempt from creep deformation at temperatures preceding the expansion of the intumescent material in fire resistance tests. The inventive glass panels have improved heat resistance, and are more easily manufactured than similar glass panels in prior art.

(57) Abrégé: L'invention est relative aux vitrages coupe-feu transparents. Le vitrage selon l'invention comprend au moins deux feuilles de verre et une couche de matériau intumescent à base de phosphates, couche située entre ces deux feuilles de verre. Le matériau intumescent comprend de la silice pyrogénée ou un mélange de silice pyrogénée et d'alumine qui confère à ce matériau notamment une absence de fluage aux températures qui précèdent l'expansion du matériau intumescent dans les épreuves d'essai au feu. Les vitrages selon l'invention présentent à la fois une résistance thermique améliorée, et se prêtent à une fabrication plus aisée que celle des vitrages analogues antérieurs.

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#5

Declaration, Power of Attorney and Petition

WE (I) the undersigned inventor(s), hereby declare(s) that:

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

TRANSPARENT FIRE BREAK GLAZING

the specification of which

- ☐ is attached hereto.
- ☒ was amended on 15 MARCH 2002
- ☒ was filed as PCT international application

Number PCT/EP00/08948on 11 SEPTEMBER 2000

and was amended under PCT Article 19

on _____ (if applicable).

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application(s)

Application No.	Country	Day/Month/Year	Priority Claimed			
9900619	BELGIUM	16 SEPTEMBER 1999	<input checked="" type="checkbox"/>	Yes	<input type="checkbox"/>	No
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			<input type="checkbox"/>	Yes	<input type="checkbox"/>	No

We (I) hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or §365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

Application Serial No.

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⑧ And we (I) hereby appoint Steven B. Kelber, Reg. No. 30,073; Jerold I. Schneider, Reg. No. 24,765; Paul C. Kimball, Reg. No. 34,641; Wilburn L. Chesser, Reg. No. 41,668; James M. Heintz, Reg. No. 41,828; Amy L. Miller, Reg. No. 43,804; Lisa K. Norton, Reg. No. 44,977; and Christopher W. Raimund, Reg. No. 47,258, as our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to Supervisor, Patent Prosecution Services, Piper Marbury Rudnick & Wolfe LLP, 1200 Nineteenth Street, N.W., Washington, D.C. 20036-2412.

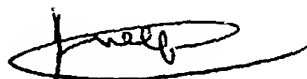
We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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Residence: Avenue des Noisetiers 66

B-1170 Bruxelles, BELGIUM

Citizen of: BELGIUM

Post Office Address: Same as above.

400 Mehdi GHODSI

NAME OF FOURTH JOINT INVENTOR

Signature of Inventor

Ghods

Date

22/03/02

BEX